Characterization and Evaluation of Materials for the US-USSR MHD Program

Quarterly Report for the
Period January-March 1976

J. Lambert Bates (Principal Investigator)

**Prepared for the Energy Research
and Development Administration
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PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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CHARACTERIZATION, EVALUATION, AND DEVELOPMENT
OF MATERIALS FOR THE US-USSR MHD PROGRAM

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ABSTRACT

Characterization and development of electrode and insulator materials are being performed in support of the International US-USSR MHD Power Generation Program. MgO insulators and five zirconia-base electrodes containing CeO_2 and Y_2O_3 additions were examined before and after Phase I testing in the USSR's U-02 MHD generator. Characterization includes thermal diffusivity/conductivity, thermal expansion, composition and microstructure. Microstructural and scanning microscopy evaluations of the 100 hour, Phase I test in USSR's U-02 are described.

1.0 OBJECTIVE

The objective of this program is to characterize, develop, and evaluate materials for the International US-USSR MHD Program and to provide technical and scientific support to the MHD materials program.

2.0 SCOPE

The scope of this program encompasses three areas:

- Provide technical assistance to MHD Division of ERDA and participating contractors with respect to selection, fabrication, and characterization of materials for testing in USSR MHD generators.
- Assist in the planning, coordination, and monitoring of screening and channel tests and materials characterization relating to US materials testing in the USSR's U-02.
- Characterize, develop, and evaluate materials for testing in the U-02 MHD generator and in other related screening tests. The characterization includes chemical, structural, and thermophysical property measurements.

3.0 TECHNICAL PROGRESS

The characterization and evaluation of electrode and insulator materials for the U-02 Phase I MHD test has continued[1,2], including pre-characterization of the electrodes and insulators and the evaluation of the US materials channel from the 100-hour U-02 Phase I test.

Some development of electrodes for the U-02 Phase II test has been initiated.

3.1 POSTCHARACTERIZATION OF U-02 PHASE I MATERIALS

The electrodes and insulators from the Phase I test of a US channel in the USSR's U-02 are being characterized and evaluated in cooperation with the National Bureau of Standards. After completion of the 127-hour test in October 1975, the channel was removed from the U-02, visually examined, and within four hours was placed in plastic bags filled with an inert gas. The channel walls were dismantled at the National Bureau of Standards in Washington, D.C., where extensive visual and photographic examinations were made. The electrode walls were separated into their separate submodules and vacuum impregnated with epoxy resin. Each submodule was cut in ethylene glycol with a diamond wheel perpendicular to the electrode surface and lengthwise parallel to the gas flow. This produced cross sections of

[1] J. Lambert Bates, Characterization and Evaluation of Materials For the US-USSR Program, Quarterly Progress Report for the Period July-September 1975, BNWL-B-463 (Oct. 1975).

[2] *Ibid*, for Period October-December 1975, BNWL-2004-1 (Jan. 1976).

each electrode and insulator. Three slices, each approximately 0.5 cm thick, were cut from one-half of the electrode length. The outside slices from each submodule were retained at NBS for phase and chemical composition studies; the inside slices were returned to the USSR for post-test analysis; and the center sections were provided to Battelle Pacific Northwest Laboratories for optical, scanning microscopy, and electron microprobe analysis. The samples for microstructural analysis were sealed in plastic in an inert gas prior to shipment.

3.1.1 Sample Preparation

At Battelle-Pacific Northwest Laboratory, the electrode-insulator samples were stored in a vacuum desiccator to prevent attack by the moisture in the air. The cross sections were again impregnated with a fluid resin under vacuum. (The impregnation at NBS had not fully penetrated into the smaller cracks and pores of the electrodes and insulators.) The mounted sections were ground without the use of lubricants to expose the electrode and insulator prior to final metallographic preparation. These cross sections were then polished through a number of steps with diamond and with a final polish with MgO, all in a nonaqueous media. All of these steps were required to prevent smearing and hydration of very soft potassium compounds, i.e., K_2CO_3 , and to minimize surface pullout.

Optical photographs of the polished sections were taken at lower magnification (8X) and montages were prepared. Higher magnifications were also made of selective specific areas. Some of the cross sections have been examined by scanning electron microscopy and electron microprobe. These techniques were used to identify the locations and compositions of the phases present in the insulator and electrodes. The compositions obtained by the electron microprobe are expressed as approximate mass ratios of the elements.

3.1.2 Microstructural Examination

The following electrode pairs have been examined to date:

1103-2103	(1101) (1102-2102) (1104)
1206-2206	(1205)(1207) (1208)
1309-2309	(1310) (1311-2311) (1312)
1414-2414	(1413) (1415) (1416)
1517-2517	(1518) (1519) (1520)

The electrodes in parentheses were photographed only at lower magnifications and more detailed examinations may be performed. Detailed descriptions for each electrode pair are described.

3.1.2.1 $0.12Y_2O_3 \cdot 0.88ZrO_2$ (1100-2100 electrode pairs)

- 1101 cathode [1.15 A/cm^2 but failed after ~ 3.5 hours of operation*] see Figure 1.
 - 1102-2102 cathode-anode [short circuit with $\sim 1.0 \text{ A/cm}^2$] see Figures 2 and 3.
 - 1103-2103 cathode-anode [0.5 A/cm^2] see Figures 4 and 5.
- Cathode 1103. The electrode was extensively fragmented. Breakup occurred mainly between the larger, dense particles of the agglomerate type electrode material. This resulted in an expansion to 0.71 cm, raising the electrode surface 0.35 cm above the adjacent MgO surface (Figures 4 and 6). The surface has recrystallized, although it remains porous (area 1a, Figures 6 and 7). The cathode is essentially free of potassium.

The electrode is single phase and contains no potassium except a trace adjacent to the Pt (Figure 6). The black area below the Pt, originally the ZrO₂ cement, is very high in potassium and in many areas is almost pure potassium. The potassium extends down into the MgO with the concentration in the MgO decreasing as the distance from the electrode increases. The potassium is located in the lower density areas which surround the dense MgO particles.

The Pt separated from the electrode, with little evidence of the wire mesh remaining. Some Pt has diffused into adjacent areas of the electrode and cement. The seed has also reacted extensively with the cement, as indicated by the black areas below the cathode. The black represents high potassium concentration.

The MgO adjacent to the electrode and at the gas surface has recrystallized and is single phase with large grains and few pores (Figures 7 and 8). About 0.1-0.2 cm below this layer is another recrystallized structure of columnar-like grains. This section also contains a second phase of Zr-Ca** located in cracks and pores (Zr/Ca $\sim 17/1$). There is also a dark band of pure zirconia containing no calcium.

Below this, the MgO is free of Zr and Ca but contains some K, which increases toward the cooler parts of the insulator.

* Failure was indicated by loss of electrical continuity; length of operation refers to time at temperature, with seed and magnetic field applied.

** The composition of the phases is denoted as metals, since the microprobe cannot detect oxygen. However, the second phase is obviously the oxides of these metals. However, the potassium could be carbonate or oxide. The compositions are given in mass ratios of the metals.

Figure 1. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Cathode 1101 after ~ 3.5 hours with current density of 1.15 A/cm^2 .

Figure 2. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Cathode 1102 after 98 hours with current density of $\sim 1.0 \text{ A/cm}^2$. See Figure 3 for corresponding anode 2102.

Figure 3. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Anode 2102 after 98 hours with current density of $\sim 1.0 \text{ A/cm}^2$. See Figure 2 for corresponding Cathode 1102.

Figure 4. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Cathode 1103 after 98 hours with current density of $\sim 0.5 \text{ A/cm}^2$. See Figure 5 for corresponding anode 2103.

Figure 5. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Anode 2103 after 98 hours with current density of $\sim 0.5 \text{ A/cm}^2$. See Figure 4 for corresponding Cathode 1103.

Figure 6. Microstructure of $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ Cathode 1103. See Area 1 of Figure 4.

Figure 7. Microstructure of MgO insulator from Cathode 1103.
See Figure 4.

Figure 8. Microstructure of $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ cathode 1103 showing electrode [1a,b] and insulator [2a,b]. See Figure 7.

- Anode 2103. There is little change in the overall structure of the electrode and insulator (Figure 5). Some separation has occurred in cement causing the electrode to raise. The MgO has fractured with cracks initiating from the inside corners of the machined cavity which held the electrode.

The electrode has recrystallized to a depth of ~ 0.05 cm below the surface with two second phases (Figures 9 and 10). The minor phase at the grain boundaries is composed of Al, K, and Ca with ratios of Al/K ~ 6 and Al/Ca ~ 10 (Figure 10-1a). This phase extends only to a depth of 0.02 cm from the surface. The lighter colored continuous phase is composed of Zr and Y, and is free of potassium. The center of the electrode has a single phase Zr/Y structure similar to the reference material but is somewhat fractured. However in some isolated areas, the lower density porous oxide areas contain varying amounts of K and Ca. One area, for example, has Zr/K ~ 2.5 and Zr/Ca ~ 7 .

The electrode immediately adjacent to the Pt and cement contain some K and some second phase with a Ca/Zr ~ 3 (Figures 9 and 10-1d).

The large ZrO_2 particles in the cement have a Zr/Ca ratio of 10. The smaller particulates surrounding these larger particles contain K with a different Zr/Ca ratio of 3.4. The Zr/K ratio is ~ 7.3 . The potassium is present only in the low density portions of the cement.

The MgO below the cement contains both K and Zr with ratios Mg/K ~ 7.1 and Mg/Zr ~ 15 . The Zr and K were concentrated in the pores and cracks. The larger MgO particles are free of K. The potassium diminishes rapidly away from the cement and at 0.1 cm from the cement only a trace of K is found. At 0.2 cm no K could be detected. The MgO at the plasma surface recrystallized to a depth of 0.31 cm from a dense surface into columnar type grains with pores at the grain boundaries. This structure eventually evolves into equiaxed grains (Figure 11). Large second phase particles, up to 45 μm , are found near the surface and are composed of Zr and Ca with Zr/Ca ~ 5 (Figure 11-2a). Some of this phase persists below the surface into the columnar grains but decreased as the distance from the surface increases.

- 1104 Cathode [0 A/cm^2 at open circuit voltage] see Figure 12.

3.1.2.2 $0.18\text{CeO}_2 \cdot 0.82\text{ZrO}_2$ (1200-2200 electrode pairs)

- 1205 cathode [0 A/cm^2 at open circuit voltage] see Figure 13.
- 1206-2206 cathode-anode [short circuit with $\sim 1 \text{ A/cm}^2$] see Figures 14 and 15.

Figure 9. Microstructure of $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ anode 2103.
See Figure 5.

1a

1b

1c

1d

Figure 10. Microstructures of $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ anode 2103. See Figure 9.

Figure 11. Microstructures of MgO insulator from anode 2103. See Figure 5.

Figure 12. $0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$ cathode 11Q4 after 98 hours with open circuit voltage, 0 A/cm^2 .

Figure 13. $0.18\text{CeO}_2 \cdot 0.82\text{ZrO}_2$ Cathode 1205 after 98 hours with open circuit voltage, 0 A/cm^2 .

Figure 14. $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ Cathode 1206 after 98 hours with current density near $\sim 0.5 \text{ A/cm}^2$. See Figure 15 for corresponding anode 2206.

Figure 15. $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ Anode 2206 after 98 hours with current density near $\sim 0.5 \text{ A/cm}^2$. See Figure 14 for corresponding Cathode 1206.

- Cathode 1206. The electrode had broken, segmented, and swollen extensively and has interacted with the cement and MgO insulator (Figures 14 and 16). The top of the 0.67 cm thick electrode has recrystallized to a depth of 0.2 cm into a single phase, dense body (Zr/Ce $\sim$ 3) with large rounded pores near the surface (Figures 16 and 17a,b). This also contains a trace of a second phase with a Zr/Ca ratio of 15. The electrode is free of this phase 0.24 cm below the surface and is also free of K except in the area immediately adjacent to the Pt (Figures 16 and 17c). The area near the Pt contains a second phase with a general composition of Zr/Si $\sim$ 3.5, Zr/K $\sim$ 2.4, and K/Ce $\sim$ 3.2

The reacted area of the electrode adjacent to the MgO and the cement is three phase and composed of fine grains and small pores (Figure 17-1d. These phases have yet to be determined but appear to have a high concentration of K.

The MgO surface, exposed to the plasma, recrystallized to a depth of $\sim$ 0.33 cm and contained two phases of Zr-Ca and Si-Ca (Si/Ca $\sim$ 5). These precipitates, 1 to 5 μ m in diameter, are found only near the surface. Immediately below this area, 0.13 cm thick columnar grains have formed (Figure 14). The grains contain some second phase material at the pores with a composition similar to that at the surface. The cement and MgO below the electrode have reacted extensively with the potassium. The resultant phase has penetrated between the platinum and the electrode. In addition, some areas are pure potassium.

- Anode 2206 (Figure 15). The electrode had recrystallized only near the plasma surface, while the remainder of the electrode had changed relatively little (Figure 18). Some breakup was observed in the lower density areas surrounding the larger particles. The electrode surface which had been exposed to the plasma, contains three phases (Figure 19-1a) one of which can only be resolved above 1000X. The major phase (gray) contains Mg, Al, and Ca with Al/Mg $\sim$ 3 and Al/Ca $\sim$ 1. Inside this gray area, there is a very fine phase (light) containing Al, Zr, and Ca with Al/Zr $\sim$ 16 and Al/Ca $\sim$ 3.5. The lighter phase, which surrounded these two phases, is Zr/Ca $\sim$ 10.4. This surface layer contained little, if any, Ce but was high in Ca which decreases rapidly away from the surface. At $\sim$ 0.3 μ m from the surface, Ca is gone and Ce has returned to the original composition. This area is free of potassium. The remainder of the anode is single phase (Figure 19b). However, the electron microprobe results indicate the presence of potassium $>$ 0.24 cm below the surface. The K occurs in several forms, one of which is a finger-like structure that often crosses some cracks, indicating it may have resulted during polishing. Another form of the potassium appears as a particle, 3-10 μ m in size, sitting on the surface. In the areas between this debris no potassium is detected. Thus, the presence of K in the lower portion of the electrode will require further evaluation. However, since all other areas show the presence of relatively little K, the source, other than from the anode itself, cannot be defined.

Figure 16. Microstructure of $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ Cathode 1206 (Area 1 in Figure 14).

1a

1b

1c

3

Figure 17. Microstructure of $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ Cathode 1206 showing areas 1a,b,c of Figure 16 and area 3 of Figure 14.

Figure 18. Microstructure of $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ anode 2206, area 1 of Figure 15.

1a

1b

1c

1d

Figure 19. Microstructure of $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ anode 2206 and cement (d).
See Figure 18.

The MgO, immediately below the cement, contains Zr and K, Mg/Zr ~ 27 and Mg/K ~ 11 . These elements are found only within a 0.05 cm band in the MgO adjacent to the cement.

The MgO exposed to the plasma has recrystallized to a depth of ~ 0.2 cm. A second phase composed of Zr and Ca is found in this area, Zr/Ca ~ 9 . The MgO surrounding this phase contains only a trace of iron, Mg/Fe ~ 30 . This area also contains no potassium. Below 0.2-0.25 cm, the potassium concentration in the MgO gradually increases, especially in the cracks. Below the large crack, near the bottom of the electrode, there are areas of high potassium in the low density MgO. Concentrations of K in the anode are small compared to corresponding areas in the cathode.

The cement contains significant amounts of potassium, although the concentrations are orders of magnitude less than at the corresponding cathode. The K is located in the lower density areas (Zr/K ~ 11) surrounding the larger calcia stabilized zirconia particles, Zr/Ca ~ 6 .

- 1207 Cathode. [$\sim 1.0 \text{ A/cm}^2$ for 3 hours then open circuit as the 2207 anode connections failed.] see Figure 20.
- 1208 Cathode. [$\sim 1.25 \text{ A/cm}^2$ for 4 hours then open circuit for 94 hours when anode 2208 connection failed.] see Figure 21.

3.1.2.3 $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ (1300-2300 electrode pairs)

- 1309-2309 cathode-anode (98 hours at $\sim 1.25 \text{ A/cm}^2$) see Figures 22 and 23.
 - Cathode 1309. This cathode had fractured more than any other of the cathodes with total separation of the electrode from the insulator (Figure 22). Sections had to be replaced in some locations and may be misoriented. This electrode has received an intensive examination because of the prolonged operation at the highest current density.

The cathode was fractured and the microstructure of the entire electrode was completely changed from that of the original microstructure. The surface recrystallized into a high porosity, but dense oxide (Figure 24). A crack extends across the entire width of the electrode, parallel to and 0.2 cm below the plasma-electrode surface. The electrode on both sides of this crack is recrystallized.

A small part of the electrode was partially detached from the surface (not shown). This area is single phase but slightly depleted in Ce with only a trace of potassium. The remaining cathode surface, also large grain and recrystallized, is also depleted in Ce (Zr/Ce ~ 3) (Figure 25-1a) and the immediate surface has a minor second phase, areas 1 and 2 (Figure 25-1b), containing magnesium and aluminum. Neither the major or second phases contain potassium.

Figure 20. $0.18\text{CeO}_2 \cdot 0.88\text{ZrO}_2$ Cathode 1207 after ~ 3 hours at $\sim 1.0 \text{ A/cm}^2$ and ~ 95 hours at open circuit.

Figure 21. $0.18\text{CeO}_2 \cdot 0.82\text{ZrO}_2$ Cathode 1208 after ~ 4 hours at $\sim 1.25 \text{ A/cm}^2$ followed by 94 hours at open circuit.

Figure 22. $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ Cathode 1309 after 98 hours at $\sim 1.25 \text{ A/cm}^2$. See Figure 23 for corresponding anode 2309.

Figure 23. $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ Anode 2309 after 98 hours at $\sim 1.25 \text{ A/cm}^2$.
See Figure 22 for corresponding Cathode 1309.

Figure 24. Microstructure of $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ Cathode 1309. Area 1 of Figure 22.

ELECTRODE

1a

1b

INSULATOR

2a

2b

Figure 25. Microstructure of $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ cathode and insulator 1309. See Figures 22 and 24.

Approximately 0.1 cm from the surface the potassium begins to appear. This corresponds to the change from a large grain, recrystallized area to a structure with fine grains, some of columnar shape. This latter area extends to the bottom of the electrode and is composed of two phases totally different from that of the original electrode. The matrix is composed of about equal amounts of Zr and Ce plus K. The second phase, ~20% of the total area, is almost pure Ce with trace amounts of Zr and no K.

Near the bottom of the cathode, immediately above the Pt, there are areas which contain significant amounts of Ca in addition to the Zr and Ce. Some Hf is also present.

In a crack in the upper left corner of the electrode, there is a wide band of solidified potassium. At the interface between the side of the electrode and MgO insulator, where some cement was originally present, a new phase has developed. The structure is porous and composed mainly of Zr with substantial Hf and K and with minor amounts of Ca. The MgO adjacent to this area contains a second phase of Zr and Hf, free of Ca and K.

The MgO at the plasma surface has undergone extensive recrystallization to a depth of approximately 0.38 cm. The top surface, ~0.08 cm deep, is a dense, large grain columnar structure. Below this is an equiaxed grain layer with a columnar-like, but lower density, structure further from the surface. (Figure 25-2a, 2b).

This area contains a bright second phase of Zr with smaller amounts of Ca (Zr/Ca ~24) and Ce (Zr/Ce ~6) with some Mg and Hf (Figure 25-2a, 2b). This phase persists into the lower density columnar grains but decreases as the distance from the surface increases. In the MgO where only small amounts of recrystallization has resulted K is present, especially in the irregularly shaped pores. The K concentration increases as the distance from the surface increases, with the K located primarily at the open pores.

The cement in the area of the Pt current lead out is essentially all potassium with some small amounts of Zr, Ce, and Ca. In the MgO, the K is concentrated in the low density areas which surround the large MgO particles.

The Pt metal current lead outs are eroded and irregular. A layer of nearly pure Zr has formed between the Pt and the bottom surface of the electrode.

The Al₂O₃ spacer (Figure 27) has reacted with the potassium. The top and highest temperature section contains Al/K ~30. The K appears to be diffusing into the Al₂O₃ from the outside surfaces

Figure 26. Microstructure of MgO insulator between cathodes 1308 and 1309. See Figure 22.

Figure 27. Al_2O_3 spacers showing areas of reaction with potassium seed.
 (a) Unreacted Al_2O_3 (no K); (b) Al_2O_3 containing potassium;
 (c) Crack in Al_2O_3 .

forming distinct reaction boundaries. The K is concentrated at the pores and the reacted area is also more porous than the unreacted area which contains no K.

- Anode 2309. The electrode has fractured and the top surface (~ 0.2 cm) is missing (Figure 23). The exposed surface which remains has recrystallized into a dense body with spherical pores (Figure 28, area 1). The electrode also cracked and has separated from the cement and platinum. The cement remains relatively intact with some separation from the MgO.

The exposed surface of the electrode which has recrystallized also has a gray second phase located principally at the grain boundaries (Figure 28-1a). This phase contains Ce, K, Zr, and Al (Ce/K ~ 0.7 , Ce/Zr ~ 4 , Al/Ce ~ 6). These ratios vary but the Al and K are always high. This second phase is limited to a narrow band ~ 0.1 cm thick along the surface.

Near the center of the electrode, there appears to be a light gray phase (Figure 28-1b). However, this has the same composition as the matrix and is thought to be shallow pores or cracks. Some of the larger cracks in the electrode interior contain K, Al, and Si with ratios K/Al ~ 1 and K/Si ~ 2 .

At the surface the MgO is recrystallized to the same depth as the electrode. A second phase is found near the surface to a depth of ~ 0.05 cm. The MgO is cracked, initiated from the machined, internal corners which seats the electrode.

A dark gray zone between the platinum and the electrode contains Zr with some Ca, apparently originating from the cement (Figure 28-1c). It does not, however, contain any K.

The larger particles in the cement immediately below the Pt are Zr and Ca (Zr/Ca ~ 7). The low density areas surrounding these particles contain about equal amounts of Zr and K plus the Ca. At 0.1 cm from the top edge of the cement, the Zr/K ratio is ~ 15 and at 0.15 cm the ratio is ~ 17 .

Near the Pt on the side of the electrode between the MgO there is a phase composed of nearly 100% Zr with a trace of K. There is no Ca present. The material adjacent to the MgO in this same area contains Zr high in K.

In the MgO below the cement, the larger particles are pure MgO. The low density MgO is high in K with some Zr. The K concentration decreases rapidly as the distance from the electrode increases.

Figure 28. Microstructure of $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ anode 2309. See Figure 23.

In the recrystallized MgO near the electrode surface, there are two phases. The light gray phase occurs only near the surface and contains Zr, Ca, Ce, and Si. The ratios are Zr/Ce ~ 4 , Zr/Ca ~ 3.4 , and Zr/Si ~ 4.4 . The bright phase contains Mg and Si with Mg/Si ratio $\sim 1.2/1$. A crack in the MgO contains a 100-150 μm band of almost pure Ca.

- 1310 cathode. [$\sim 1.0 \text{ A/cm}^2$ at short circuit voltage for 98 hours] see Figure 29.
- 1311-2311 cathode-anode pair [$\sim 0.5 \text{ A/cm}^2$ for 98 hours] see Figures 30-31.

3.1.2.4 $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ (1400-2400 electrode pairs)

- 1413 cathode (0 A/cm^2 for 98 hours at open circuit voltage) see Figure 32.
- 1414-2414 cathode-anode pair ($\sim 0.5 \text{ A/cm}^2$ for 98 hours) see Figures 33-34.
- Cathode 1414. The changes in the upper portion of the electrode were extensive with the electrode separating from the Pt and cement. The MgO was cracked, but not extensively degraded (Figure 33). The top half of the electrode had fragmented into smaller pieces with only remnants of larger particles remaining. The surface had recrystallized to a depth of $\sim 0.03 \text{ cm}$.

The Pt paste and mesh had separated from the electrode and from the cement in the center and the mesh was eroded slightly.

A recrystallized band, 0.025 cm thick at the electrode surface (Figure 35), contains very small, 10-25 μm diameter, second phase particles composed of Al, Ca, and Ce, with ratios of Ce/Al ~ 7.5 and Ce/Ca ~ 12 (Figure 36-1a). This phase does not contain Zr and is found in a band extending to a depth of 300 μm from the surface.

Next to the surface is a narrow band of almost pure K. Potassium is also found in some cracks in this recrystallized area. Some pores in this area also contain a phase composed of Zr, K, and Ce with ratios Ce/Zr ~ 5 and Ce/K ~ 5 . The continuous matrix contains no potassium. In the broken central portion of the electrode (Figure 36b) the oxide contains no K with Ce/Zr ~ 5 .

The bottom of the electrode (Figure 36-1c) is high in K, especially in the low density areas surrounding the large particles and in the cracks in the larger particles. Some 1-5 μm particles of pure K are scattered in the low density area. There is a narrow band of pure K adjacent to the bottom of the electrode. The platinum is partly eroded, presumably by the K since the pores in the Pt are also high in K.

Figure 30. $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ Cathode 1311 after 98 hours with current density of $\sim 0.5 \text{ A/cm}^2$. See Figure 31 for corresponding anode 2311.

Figure 31. $0.5\text{CeO}_2 \cdot 0.5\text{ZrO}_2$ Anode 2311 after 98 hours with current density of $\sim 0.5 \text{ A/cm}^2$. See Figure 30 for corresponding Cathode 1311.

Figure 32. $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ Cathode 1413 after 98 hours with 0 A/cm^2 at open current voltage.

Figure 33. $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ Cathode 1414 after 98 hours at $\sim 0.5 \text{ A/cm}^2$. See Figure 34 for corresponding anode 2414.

Figure 34. $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ Anode 2414 after 98 hours at $\sim 0.5 \text{ A/cm}^2$.
See Figure 33 for corresponding cathode 1414.

Figure 35. Microstructure of $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ cathode 1414. See Figure 33.

1a

1b

1c

Figure 36. Microstructure of $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ cathode 1414.
See Figure 35.

The cement contains significant quantities of K located in the low density around the large solid particles. These particles contain Zr but no Ca. The Ca is located in the smaller granular phase which surrounds these particles. The MgO immediately below the cement is higher in K. However, the K diminishes rapidly from the MgO cement interface. The MgO is free of K 0.2 cm from the cement.

In the recrystallized MgO near the electrode surface, there is a high concentration of K in the pores and cracks. In addition, there is a light second phase containing Zr, Ce, Ca, and Mg with ratios $Zr/Cr \sim 10$, $Zr/Ca \sim 16$, and $Zr/Mg \sim 20$. This phase seems to be located only in the more dense bands ~ 0.1 to 0.6 cm next to the surface. Below this band, this phase is less dense and is smaller in size and the composition changes to a Ce/Zr ratio of 5.0 with no Ca or Mg.

The potassium in the MgO increases as the distance from the surface increases.

- Anode 2414 (Figure 34). The electrode and insulator are relatively intact although the electrode is badly cracked and separated from the Pt and cement (Figure 37). The electrode separated horizontally and parallel to the surface about midway between the top and the bottom. Recrystallization occurred to a depth between 0.06 to 0.1 cm from the surface.

Two phases are associated with this surface recrystallization (Figure 38-1a). The major light colored phase contains Zr, Ce, and some Ca with ratios $Ce/Zr \sim 4$ and $Ce/Ca \sim 10$. There is no K in this phase. The minor gray phase contains Al, Cr, K, Ca, and Ce with ratios $Al/Ce \sim 3$, $Al/Zr \sim 31$, $Al/K \sim 15$, and $Al/Ca \sim 5$. These phases appear in areas isolated near the surface. The electrode near the Pt contains Zr, Ca, and Ce inclusions in the pores with $Zr/Ca \sim 7$ and $Zr/Ce \sim 5$ (Figure 38-1c). This portion of the electrode is free of K.

The low density portion of the cement contains K with $Zr/K \sim 3$. This ratio varies widely within the cement. The MgO, 1 cm adjacent to the cement, contains a small amount of K. Beyond this there is little or no K.

The MgO at the surface adjacent to the electrode recrystallized area contains larger spherical pores with a bright second phase at the surface. This second phase is composed of Ce, Zr, Ca and a trace of Si with $Ce/Zr \sim 2$, $Ce/Ca \sim 5$, and $Ce/Si \sim 8$. The continuous phase in this area contains Mg with minor amounts of Zr, K, Ca, Fe, and Ni, with ratios $Mg/Zr \sim 33$, $Mg/K \sim 11$, and $Mg/Ca \sim 13$, $Mg/Fe \sim 22$, and $Mg/Ni \sim 33$. This latter phase is concentrated only at the surface and at 0.1 cm from the surface, only Si and K remain. There is a large increase of K in the cracks of the MgO.

Figure 37. Microstructure of $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ anode 2414. See Figure 34.

1a

1b

1c

Figure 38. Microstructure of $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ anode 2414. See Figure 37.

- 1415 cathode [0 A/cm^2 with open circuit voltages. This electrode failed during pre-test startup testing] see Figure 39.
- 1416 cathode [$\sim 1.24 \text{ A/cm}^2$ for 5-1/2 hours followed by 92-1/2 hours at open circuit voltage after failure] see Figure 40.

3.1.2.5 $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ (1500-2500 electrode pairs)

- 1517-2517 cathode-anode [90 hours at $\sim 1.25 \text{ A/cm}^2$ plus 8 hours at 0 A/cm^2 following current leadout failure] see Figures 41 and 42.
- Cathode 1517 (Figure 41). The electrode has experienced substantial fracture with nearly complete restructuring (Figures 41 and 43). The MgO insulator and cement supporting the electrode are extensively degraded with the Pt separated from the electrode.

The surface of the electrode exposed to the plasma has recrystallized to a depth of 0.04 cm and is two phase containing large pores (Figure 44-1a). Along the very outer edge (0.01 cm width) there is a phase containing Zr, Ce, and Ca with a Zr/Ce ratio of ~ 4 and a Zr/Ca ratio near 7. There is little or no Ta in this layer. This area of high Zr decreases very rapidly and at about 100 μm below the surface the Ce/Zr ~ 2 and the Ta begin to increase. This area is also free of K.

Beyond 0.01 cm, there are three phases, a light gray phase containing Ce, Ta, Ca, and Zr with Ce/Ta ~ 4 ; Ce/Ca ~ 4 ; and Ce/Zr ~ 3 (Figure 42-1a). The darker phase contains Ce, Ca, and Zr with ratios Ce/Ca ~ 3.5 and Ce/Zr ~ 6 . The area is also free of K. The original continuous phase has a Ce/Zr ~ 12 with a trace of Ta. At approximately 0.1 cm beneath the surface, the Ce/Zr/Ta ratios approach that of the reference electrode. At 0.25 cm beneath the surface, K begins to appear with a Ce/K ratio ~ 12 . At 0.35, 0.45, and 0.55 cm, the surface of the Ce/K ratios are approximately 8, 6, and 6 respectively.

In the center of the electrode is a second phase comprising 1-2% of total area (Figure 44-1b). The continuous phase is Ce and Zr only with Ce/Zr ~ 12 . The second phase contains Ce, K, Zr, and a trace of Ta with Ce/K ~ 12 , Ce/Zr ~ 3 . This structure extends down to the bottom of the electrode.

Between the separated Pt and the electrode is a large porous structure area containing long needle-like grains (Figure 44-2a). This area is almost pure Zr and is free of Ce, Ca, but does contain some K. The black area below the Pt is very high in K (Figure 44-3a). The remnants of the cement contain large ZrO_2 particles with Zr/Ca ~ 8 . The area surrounding these particles, which originally were low density cement, is nearly pure K.

Figure 39. $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ cathode 1415 after 98 hours with 0 A/cm^2 at open circuit voltage.

Figure 40. $0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$ Cathode 1416 after 5-1/2 hours at $\sim 1.25 \text{ A/cm}^2$ and 92-1/2 hours at 0 A/cm^2 .

Figure 41. $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ cathode 1517 after 90 hours at $\sim 1.25 \text{ A/cm}^2$ and 8 hours at 0 A/cm^2 . See Figure 42 for corresponding anode 2517.

Figure 42. $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ anode 2517 after 90 hours at ~ 1.25 A/cm² and 8 hours at 0 A/cm². See Figure 41 for corresponding anode 1517.

Figure 43. Microstructure of $0.8\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ cathode 1517. See Figure 42.

ELECTRODE

1a

1b

2a

3

Figure 44. Microstructure of $0.8\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ cathode 1517.
See Figure 43.

The MgO below the cement contains large quantities of K. The Mg/K ratios at the cement-MgO interface and 0.1 cm below are 4 and 3 respectively.

The MgO at the plasma surface has also restructured. Immediately adjacent to the surface is a single phase recrystallized area, some of which appears to have spalled away. This area contains Ce, Ca, K, Zr, and Ta with ratios $Ce/Ta \sim 12/1$, $Ce/Ca \sim 5$, $Ce/K \sim 6$, and $Ce/Zr \sim 2$. This apparently came from the electrode and became attached to the insulator.

Immediately below this begins a two phase region which at a depth of 0.2 cm contains pure Mg and a grain boundary, minor phase containing Ce, K, and Zr, with ratios $Ce/K \sim 1.6$ and $Ce/Zr \sim 1$. This second phase decreases toward the bottom of the electrode and the potassium begins to increase, especially at the grain boundaries.

- Anode 2517. The electrode has fractured considerably (Figure 42). However, this was an instrumented section and the central hole may have contributed to the fragmentation. The surrounding MgO is also cracked with most structural changes occurring near the plasma surface. The Pt has separated from the electrode while the cement remains partially intact.

The surface of the anode has recrystallized to a depth of 0.25-0.3 cm, (Figure 45.) Along the entire surface is a two phase area with a gray phase containing Al and K with $Al/K \sim 3.4$ and a light phase containing Zr, Ca, and Ce, with $Ce/Ca \sim 4$ and $Ce/Zr \sim 1$, (Figure 46-1a). This is much lower than normal. The concentration of this phase decreases rapidly toward the interior of the electrode and at 0.05 cm below the surface the Zr/Ce ratio has returned to the original value.

The remainder of the electrode has a normal composition, (Figure 46-1b) but small amounts of K are found in the cracks.

The cement contains high concentrations of K located in the low density porous areas of the cement.

The MgO immediately adjacent to the cement contains considerable K and also some Zr, with the $Mg/K \sim 7$ and the $Mg/Zr \sim 14$. Both the Zr and K are located in the lower density MgO and decrease rapidly in concentration toward the bulk MgO. At 0.1 cm below the cement, the $Mg/K \sim 13$ and at 0.4 cm the MgO is free of K.

The MgO surface has recrystallized to varying degrees to depths up to 0.4 cm. This area is generally single phase MgO except at the surface where some two phase particles are attached. The lighter phase contains Zr and Ca only with Zr/Ca ratio of ~ 8 . The darker continuous gray phase contains Al, Mg, and traces of Fe. The

Figure 45. Microstructure of $0.8\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ anode 2517. See Figure 42.

ELECTRODE

1a

1b

ELECTRODE

1c

INSULATOR

2a

Figure 46. Microstructure of $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ anode and insulator 2517. See Figure 45.

Al/Mg ratio is ~ 2.8 and the Al/Fe ratio is ~ 19 . This area is free of K. Another particle contains only Zr and Ca with Zr/Ca ratio of 9. Some of these phases extend into the MgO to a depth of ~ 0.05 cm.

The MgO contains only small amounts of K but this concentration increases as the distance from the surface increases. Only trace amounts of K are found near the surface and at 0.18 and 0.35 cm below the surface, the Mg/K ratios are 20 and 4 respectively. Below the large crack which is found ~ 1 cm below the surface, the K again begins to decrease.

- 1518-2518 cathode-anode [98 hours at 1 A/cm^2 short circuit] Figure 47.
- 1519 cathode [98 hours at 0.5 A/cm^2] Figure 48.
- 1520 cathode [98 hours at 0 A/cm^2 open circuit voltage] Figure 49.

3.1.3 General Conclusions of Metallography and Electron Scanning and Microprobe Analyses

The following conclusions were made from the metallographic and electron microscope evaluations of the electrodes described previously. Since the examination and evaluations are continuing, these conclusions may be modified.

3.1.3.1 General

- The cathode degradation was substantially greater than the anode, presumably due to electrochemical effects.
- Cement, which acts as a source of potassium concentration, is a major design problem.
- Potassium transport and concentration occurs principally through and in the open porosity of the low density phases of the MgO, cement and electrode.
- The driving force for potassium transport appears to be the high electrical current density and voltage, causing the potassium ions to migrate to the cathode.
- The swelling of the cement and MgO appears to result from hydration of K_2CO_3 which is accompanied by an approximate 40% increase in volume.
- Microstructural and electron microscopy results agree in general with the x-ray diffraction phase studies performed by the National Bureau of Standards.

Figure 47. $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ cathode 1518 after 98 hours at 1 A/cm^2 .

Figure 48. $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_3$ cathode 1519 after 98 hours at $\sim 0.5 \text{ A/cm}^2$.

Figure 49. $0.80\text{CeO}_2 \cdot 0.18\text{ZrO}_2 \cdot 0.02\text{Ta}_2\text{O}_5$ cathode 1520 after 98 hours at 0 A/cm^2 open circuit voltage.

- Contamination of the insulator and electrode surfaces are apparently due to oxides carried by the plasma to the electrodes from other components of the MHD generator, i.e., the combustor.
- There is a preferential loss of Ce and/or Ta from the surfaces of the high Ce containing electrodes.
- Potassium not only influences the mechanical breakup of the MgO, cement and electrodes, but also appears to influence or enhance recrystallization, transport of other elements, and formation of new phases in the electrodes and insulators.

3.1.3.2 Potassium

The highest concentrations of potassium are associated with the cathode side of the channel, concentrating in the materials which were at operating temperatures between approximately 600 and 1300°C. In the MgO and cement, the potassium is associated with the lower density, porous areas which surround the large oxide particles. Potassium is also found in cracks. Little potassium is found on or near the plasma surface of the MgO or cathode. Some potassium was found on the anode surface and in the cement around the anode.

The transport of potassium is directly related to the charge and density of the electrical current and to the porosity of the electrode and insulator. The potassium as an ion, apparently is attracted to the cathode by the negative charge and is transported into oxides by the current flow and voltage. The paths of easiest movement appear to be the open pores. Once present, and in higher concentrations, it can more readily react with the electrode and insulator.

3.1.3.3 MgO Insulator

The MgO exposed to the plasma has recrystallized to depths as much as 0.4 cm. Although not observed in all electrode pairs, the recrystallization of the MgO generally consists of three different structures.

- (1) Dense band near the surface which varies in width from 0.02 to 0.10 cm. This layer is 3 to 5 times wider on the cathode side than on the anode. This layer also contains a second phase composed principally of zirconia and calcia. However, on the downstream electrodes, this phase also contains increasing amounts of cerium. The calcium concentration is highest on the anode. The anode also often contains silicon. No potassium is observed at the surface. Other impurities, e.g. Fe, Ni, Al, are also observed on specific electrodes, but in small amounts.

- (2) Columnar type grains extend to depths between 0.7-0.18 cm. (The depths are approximately the same for the cathode and anode sides of the channel). These long grains are associated with a high porosity at the grain boundaries. On the cathode side, a second phase is found at the grain boundaries. This phase is thought to be a CaO stabilized ZrO₂. The CaO/ZrO₂ ratio increases toward the downstream part of the channel. Little, if any, second phase is present in the columnar grains of the MgO near the anode. This area, except in selected cracks, is also free of potassium.
- (3) Equiaxed grain growth occurred to a depth from the surface of approximately 0.3 to 0.4 cm. This recrystallization appears as a densification of the low density fines which surrounded the larger particles, in a decrease in the number of pores, and in an increase in pore size. This recrystallized area of the cathode sometimes contains a second phase and is also the area where potassium is first observed in the MgO.
- The erosion, degradation, and recrystallization of the MgO near the plasma surface appears to be more severe on the MgO which is on the upstream side of the electrode. Perhaps associated with this effect is the apparent increased reaction between the MgO, the cement, and the upstream side of the electrode. This effect appears to be more pronounced on the anode than on the cathode.
 - The cracking of the MgO appeared similar for all electrodes. Cracks radiated out from and appeared to initiate from the corners of the machined recesses in the insulator. In most insulators, these cracks were started at ~0.6-0.7 cm from the surface. Most of these cracks contained potassium.
 - The potassium penetrated to extreme depths in the MgO below the cement on the cathode side. Penetration in the MgO below the cement of the anodes is minor, with only trace amounts 0.05 cm below the cement-MgO interface. The potassium penetration is greatest in the high current density cathodes.

3.1.3.4 Electrodes

The microstructure of each electrode pair is unique but some general conclusions may be made.

- The surface of most electrodes has recrystallized into a dense layer, often with large spherical pores. This layer varies in thickness (0.02-0.3 cm) between electrode materials. With the exception of the 1103 cathode, the surface contains at least one and up to three phases, with the concentration of the second phases highest nearest the plasma surface. These second phases

Table I. PHASES AND APPROXIMATE COMPOSITIONS IN ELECTRODES: NEAR
PLASMA SURFACE

<u>Cathode</u>	<u>MAJOR PHASE*</u>	<u>MINOR PHASE*</u>
1103	Zr/Y ~2.6	
1206	Zr/Ce ~3	(1) Zr/Ca ~15
1309	Zr/Ca	
1414		(1) Ce/Al ~7.5, Ce/Ca ~12
1517	(1) Ce/Zr ~12 (Cont.)	(1) Ce/K ~12, Ce/Zr ~3
<u>Anode</u>		
2103	Zr/Y ~2.6	Al/Ca ~10, Al/K ~6
2206	Zr/Ca ~10.4 (at surface)	(1) Al/Mg ~3, Al/Ca ~11
	Zr/Ce ~3 (>0.3 μm)	(2) Al/Zr ~16, Al/Ca ~3.5
2309	Zr/Ce ~0.2	(1) Ce/K ~0.7, Ce/Zr ~4, Al/Ce ~5.5
2414	Ce/Zr ~4.5	(1) Ce/Zr ~4, Ce/Ca ~10
		(2) Al/Ce ~3, Al/Zr ~31, Al/K ~15, Al/Ca ~5
2515	Ce/Zr ~1, Ce/Ca ~4 (>0.025 cm) Ce/Zr ~7 (>0.025 cm)	(1) Al/K ~3.4

* The compositions are expressed in mass ratios of the metals.

on the cathode are principally zirconia containing varying amounts of calcium and cerium. On the anode surfaces, more than one phase is found and in addition to the zirconia-calcia-ceria phase, phases high in aluminum are present. Potassium is also found in some phases. These results are summarized in Table I.

The phase compositions at the center of the electrodes also varies, and often contain more than one phase. These results are summarized in Table II.

Table II: PHASES AND APPROXIMATE COMPOSITIONS IN ELECTRODES: NEAR
CENTER

	<u>MAJOR PHASE*</u>	<u>MINOR PHASE*</u>
<u>Cathode</u>		
1103	Zr/Y ~2.6	
1206	Zr/Ce ~2.6	(1) Zr/Ca ~11 to no Ca
1309	Zr/Ce ~1, Zr/K ~0.5	(1) Ce (20%)
1414	Ce, Zr, K	(1) K (in cracks and pores)
1517	Ce/Ta ~4, Ce/Ca ~4, Ce/Zr ~3	(1) Ce/Ca ~3.5, Ce/Zr ~6. (2) Zr/Ce ~4, Zr/Ca ~7
<u>Anode</u>		
2103	Zr/Y ~2.6	(1) Zr/K ~2.5, Zr/Ca ~7 (in pores and cracks)
2106	Zr/Ce ~2.6	(1) K (high in low density oxide)
2309	Zr/Ce ~0.2	(1) K/Al ~1, K/Si ~2 (in cracks)
2414	Ce/Zr ~4.5	
2517	Ce/Zr ~7	(1) K (trace in cracks)

*The compositions are expressed in mass ratios of the metals.

Similarly, the electrode near the platinum current leadout was also altered. The possible phases and compositions are summarized in Table III.

Table III. PHASES AND APPROPRIATE COMPOSITIONS IN ELECTRODE: ADJACENT PT LEADS

	<u>MAJOR PHASES*</u>	<u>MINOR PHASES*</u>
<u>Cathode</u>		
1103	Zr/Y ~2.6	(1) K
1206	Zr/Ce ~3.2	(1) Zr/K ~2.4 (2) Zr/Si ~3.5
1309	(Zr+Ce)/K ~1	(1) K (in pores)
1914	Ce, Zr, K	(1) K
1517	N.D.	N.D.
<u>Anode</u>		
1103	Zr/Y ~2.6	(1) Zr/K ~9, Zr/Ca ~1.3
1206	Zr/Ce ~3	(1) K
2309	Zr/Ce ~0.2	(1) Pt (2) Zr/Ca
2414	Ce/Zr ~4.5	(1) Zr/Ce ~5, Zr/Ca ~7
2517	Ce/Zr ~7	(1) K

*The compositions are expressed in mass ratios of the metals.

3.1.3.5 Cement

The cement acts as a major source of potassium concentration and possible path of seed transport into the MgO and electrode. The highest concentration of potassium in the cement is associated with the cathodes and increases with current density. Total degradation of the cement can occur. Conversely the cement associated with the anode remains intact, but contains ~10% potassium associated totally with the low density portion of the cement.

The cement initially contains phosphorus as a binder and curing agent. However, because of interference with the presence of zirconium on the electrode and cement, the electron microprobe cannot detect or evaluate its presence or location. Therefore, its effect on the results cannot be evaluated.

3.1.3.6 Aluminum Oxide

The Al_2O_3 spacers reacted with the potassium, but did not appear to react in any other way with the other materials. The higher temperature portion of the sintered Al_2O_3 plate appeared to form $\beta\text{-Al}_2\text{O}_3$ with $\text{Al/K} \sim 30$. The potassium diffuses into the Al_2O_3 from the outside edges forming a distinct cone-shaped reaction boundary. The potassium is concentrated at pores within the Al_2O_3 . The $\beta\text{-Al}_2\text{O}_3$ has a higher porosity than the original Al_2O_3 .

4.0 WORK ANTICIPATED DURING NEXT QUARTER

4.1 CHARACTERIZATION OF U.S. MHD CHANNEL TEST IN U-02, PHASE I

Some additional characterization of the samples from the US U-02 Channel, Phase I, will be made and evaluated. The distribution of the potassium both in some electrodes and insulators, from the overall distribution along the temperature gradient, to the microscopic distribution in pores, grain boundaries and grains. Similar scans will be made for Ce and Ta.

4.2 THERMAL PROPERTIES

The thermal conductivity of the electrodes will be calculated using measured thermal diffusivity, density and thermal expansion data, and calculated heat capacity values.

4.3 ELECTRODE DEVELOPMENT

The development of controlled microstructures for $\text{CeO}_2\cdot\text{ZrO}_2$, $3\text{MgO}\cdot\text{Al}_2\text{O}_3\cdot\text{Fe}_2\text{O}_4$, La(Mg) CrO_3 and $\text{FeO}\cdot\text{Al}_2\text{O}_3$ (Fe_3O_4) will be initiated.

Some methods for grading electrode composition and applying metal surfaces will be evaluated.

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